PROJECT NUMBER: 6505
PROJECT TITLE: Special Investigations and Methods Development
PROJECT LEADER: W. R. Harvey
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I. Metal Determinations

Cadmium determinations continue on samples of grafted tobacco and also normal leaf samples in support of the cadmium uptake study. A microwave digestion oven from the SEM company is being evaluated for our digestion needs. Up to 12 samples can be digested at one time using nitric acid and hydrogen peroxide. Preliminary results are encouraging.

II. Cooked Flavor Support

A. Precook by Continuous Flow

Both glass and stainless steel coils plugged with the precook when left standing for 4 days at 95°C. No restrictions were in either of the coils.

B. HPLC Examination of Cooked Flavors

Best separation of cooked flavor constituents was achieved using an HP diol 200mm x 4.6mm column with a water-methanol gradient. More than 20 peaks were detected in the 200-60nm range. These peaks showed similar spectra with a primary absorption maxima at 272-274nm. Extraction with methylene chloride had no effect on the magnitude of these peaks. This indicates a large group of polar "azines" e.g. pyrazines. The two largest peaks were identified as 2,5 and 2,6 deoxyfructosazine. No additional information was obtained when the cooked flavor effluent from the Dionex ion chromatograph was examined by the HPLC detector.

C. IC Scans for Cooked Flavors

IC scans were done on six samples of cooked flavors. These scans are now being done by General Analytical.

III. Volatile Acid Determination in RL Sheet

In the development of an analytical procedure for acetic and formic acid in RL sheet, an apparent buildup of volatile acids was observed when RL was packed in gelatin capsules over a period of time (4 days). An attempt to repeat this with the same samples was not successful. The reasons for this are under investigation since autoclaving the samples would have indicated whether or not the volatile acid buildup was of microbiological origin.

IV. Menthol Project

A total of eight cigarette filler samples were examined by ion chromatography. Anions and monovalent and divalent cations were determined.

V. X-Ray Flourescence Analysis (XRF)

Samples of lower stalk position tobacco leaves were ground in the ball mill and the sand isolated by nitric acid digestion. Particle size will be determined by Project 1720. Variation in particle size is a drawback to determination of sand (as Si) in tobacco leaf by XRF and it is hoped that the ball mill will reduce the sample to a more homogeneous state. SiO_2 added to bright monitor tobacco as standards in the 0.5 to 10% range shows scatter, probably due to lack of homogeneity. The pure element reference files for Si and interfering elements have been established. The quantitative standards file has been set up.

VI. Reports

1. Palmer G. Baker and William R. Harvey, Special Report, "The Determination of Acetic and Formic Acids in RL Sheet by Ion Chromatography," Accession Number 85-217, November, 1985.

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